

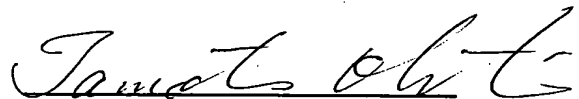
VERIFICATION OF TRANSLATION

The undersigned translator, having an address at Bridgestone Toranomon Building 6F, 25-2, Toranomon 3-chome, Minato-ku, Tokyo, 105-0001 Japan declares that:

- (1) I am fully conversant both with the Japanese and English language.
- (2) I have translated into English the Japanese Patent Application No. 2000-157802 filed on May 29, 2000. A copy of said English translation is attached hereto.
- (3) The translation is to the best of my knowledge and belief, an accurate translation of the application as filed into the English language.

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief believed to be true.

Date: September 9, 2003



Tamotsu Ohtani

PATENT OFFICE
JAPANESE GOVERNMENT

This is to certify that the annexed is a true copy of the following application as filed with this office.

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Application Number: Patent Application No. 2000-157802

Applicant: Idemitsu Petrochemical Co., Ltd.

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Commissioner,
Patent Office

Kozo Oikawa

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[Reference No.] N00-0041

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[Title of the invention]

Polycarbonate resin for optical disk substrates and the optical disk substrate

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[List of Item Filed]

[Item] Specification 1

[Item] Abstract 1

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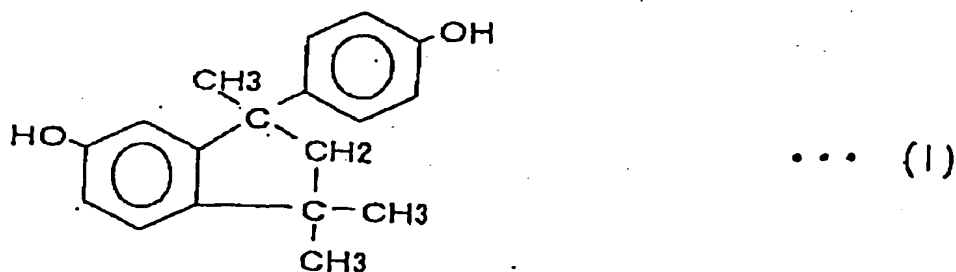
[Requirement of Proof] Yes

[Document] Specification

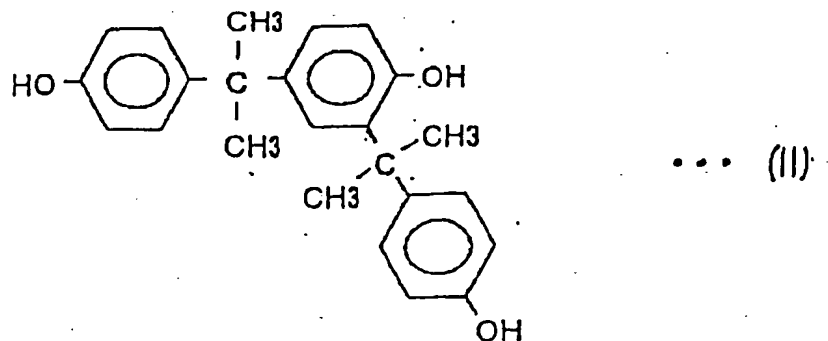
[Title of Invention] POLYCARBONATE RESIN FOR OPTICAL DISK
SUBSTRATE AND OPTICAL DISK SUBSTRATE

[Claims]

[Claim 1] A polycarbonate resin for an optical disk substrate characterized by being produced with, as a raw material, 2,2-(4-hydroxyphenyl)propane having a content of 2-(2-hydroxyphenyl)-2-(4-hydroxyphenyl)propane of 1,000 ppm or less, a content of a cyclic dimer of p-isopropenylphenol represented by the following general formula (I) of 150 ppm or less:



and a content of a trisphenol compound represented by the following general formula (II) of 150 ppm or less:



having a viscosity average molecular weight of from 10,000 to 17,000 and a fraction of hydroxyl end groups of less than 7% by mole, and containing from 100 to 500 ppm of a releasing agent.

[Claim 2] A polycarbonate resin for an optical disk substrate as described in claim 1, which has a free total phenol content of 80 ppm or less.

[Claim 3] A polycarbonate resin for an optical disk substrate described in claim 1 or 2, which contains from 150 to 350 ppm of a releasing agent.

[Claim 4] A polycarbonate resin for an optical disk substrate described in any of claims 1 to 3, wherein the releasing agent is a polyhydric alcohol fatty acid ester.

[Claim 5] An optical disk substrate comprising a polycarbonate resin for an optical disk substrate described in any of claims 1 to 4.

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs]

The present invention relates to a polycarbonate resin for an optical disk substrate and an optical disk substrate, and more particularly, it relates to a polycarbonate resin that is suitable for a material of an optical disk substrate having less defects due to flash, and an optical disk substrate formed with the resin.

[0002]

[Description of the Prior Art] A polycarbonate resin is widely used as a substrate of an optical disk, such as CD, CD-ROM, MO, CD-R, CD-RW, DVD-ROM, DVD-R and DVD-RAM, owing to the transparency, the heat resistance and the low water absorbing property thereof. In general, these optical disks are often produced by such a process that a surface formed by engraving signals, such as pits and grooves, on a thin plate of nickel called as a stamper arranged in a metallic mold is transferred to a substrate formed with a polycarbonate resin through injection molding.

[0003] In the production of a substrate of an optical disk in recent years, several tens to a number exceeding one hundred of molding machines are introduced in one factory to further enhance the mass production volume efficiency, and simultaneously quality control is severely ensured. However, although there is no change of a stamper or no change in molding conditions, and the factors determining the flowability of a polycarbonate resin, such as the molecular weight, the molecular weight distribution, the glass transition temperature and the like, are stable within prescribed ranges, there are some cases where releasing failure extemporaneously occurs upon sprue cutting of a center hole of a disk substrate, and the outer circumference of the disk substrate or fine flash formed on the outer circumference is in friction with a metallic mold due to the releasing failure to cause contamination of resin dusts, whereby the yield of the disk substrates is decreased to several percents

to several tens percents. The phenomenon is generally referred to as a defect due to flash, which can be prevented by increasing an amount of a releasing agent. However, the amount of the releasing agent cannot be increased to an unnecessary level because there is a tendency of occurrence of defects on optical characteristics, particularly such a tendency that occurrence of defects of polarization and white turbidity in the polycarbonate resin is accelerated upon an accelerated aging test under constant temperature and humidity, whereby the reliability of storage stability of recorded data as an optical disk is also lowered.

[0004]

[Problem(s) to be Solved by the Invention]

The invention has been developed under the circumstances, and is to provide a polycarbonate resin that is suitable for a material of an optical disk substrate having less defects due to flash without increase of an addition amount of a releasing agent to an unnecessary level, and an optical disk substrate formed with the resin.

[0005]

[Means for Solving the Problem]

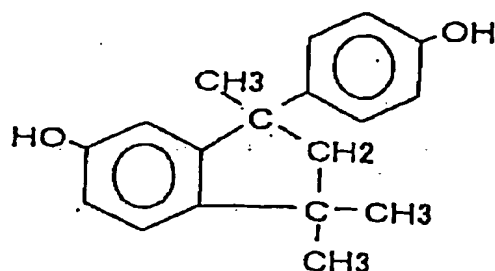
As a result of various investigations made by the inventors, it has been found that occurrence of defects due to flash is correlated with the particular impurities in 2,2-(4-hydroxyphenyl)propane as a raw material and a fraction of hydroxyl end groups in the polycarbonate resin, and thus the

first invention to the fourth invention have been completed.

[0006] That is, the summary of this invention is as follows.

1. A polycarbonate resin for an optical disk substrate characterized by being produced with, as a raw material, 2,2-(4-hydroxyphenyl)propane having a content of 2-(2-hydroxyphenyl)-2-(4-hydroxyphenyl)propane of 1,000 ppm or less, a content of a cyclic dimer of p-isopropenylphenol represented by the following general formula (I) of 150 ppm or less:

[0007]

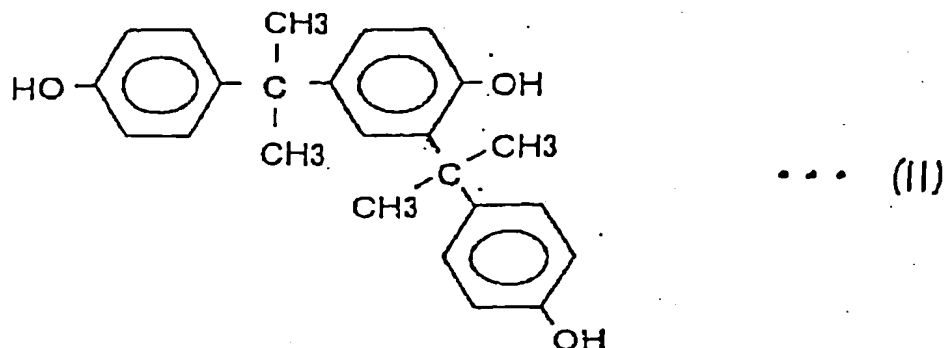


... (I)

[0008]

and a content of a trisphenol compound represented by the following general formula (II) of 150 ppm or less:

[0009]



[0010] having a viscosity average molecular weight of from 10,000 to 17,000 and a fraction of hydroxyl end groups of less than 7% by mole, and containing from 100 to 500 ppm of a releasing agent.

2. A polycarbonate resin for an optical disk substrate as described in above 1, which has a free total phenol content of 80 ppm or less.

3. A polycarbonate resin for an optical disk substrate described in above 1 or 2, which contains from 150 to 350 ppm of a releasing agent.

4. A polycarbonate resin for an optical disk substrate described in any of above 1 to 3, wherein the releasing agent is a polyhydric alcohol fatty acid ester.

5. An optical disk substrate comprising a polycarbonate resin for an optical disk substrate described in any of above 1 to 4.

[0011]

[Embodiments of the Invention] Hereafter, this invention is explained in detail. In the invention, it is necessary that the amount of 2,4-bisphenol A is 1,000 ppm or less, the amount of an IPP dimer is 150 ppm or less, and the amount of trisphenol

is 150 ppm or less in bisphenol A as a raw material of the polycarbonate resin. When the amounts each is too large, defects due to flash frequently occur upon molding the polycarbonate as a disk.

[0012] Bisphenol A herein is produced in an ordinary manner from phenol and acetone, but it is necessarily purified upon a certain step of the process. While various kinds of purification methods can be employed, it is preferred that purification is carried out in the step of an adduct of bisphenol A and phenol, as described in JP-A-7-25798. That is, in a production process of bisphenol A, in which an adduct of bisphenol A and phenol is crystallized from a phenol solution of bisphenol A obtained through a reaction of phenol and acetone in the presence of an acid catalyst, and after solid-liquid separation of the resulting slurry, phenol is removed from the solid component, it is sufficient that before removing phenol after the solid-liquid separation of the slurry, such an operation is repeated at least once that the adduct of bisphenol A and phenol is dissolved in phenol and then crystallized, followed by solid-liquid separation of the slurry.

[0013] The polycarbonate resin used in the optical disk substrate of the invention will be described.

As the polycarbonate resin, an aromatic polycarbonate resin produced through a reaction of bisphenol A as a dihydric phenol and a carbonate precursor is preferably used. The production process may be either the solution process or the molten process.

In this case, as the carbonate precursor, a carbonyl halide, a carbonyl ester and a haloformate can be used. More specific examples thereof include phosgene, dihaloformate of a dihydric phenol, diphenyl carbonate, dimethyl carbonate, diethyl carbonate and the like.

[0014] With respect to the chemical structure of the polycarbonate resin, those having a molecular chain having a linear structure, a cyclic structure or a branched structure can be used. Among these, as the polycarbonate resin having a branched structure, those produced by using, as a branching agent, 1,1,1-tris(4-hydroxyphenyl)ethane, $\alpha, \alpha', \alpha''$ -tris(4-hydroxyphenyl)-1,3,5-triisopropylbenzene, fluoroglycine, trimellitic acid, isatin bis(o-cresol) and the like are preferably used.

[0015] The viscosity average molecular weight of the polycarbonate resin is generally from 10,000 to 30,000, but it is necessarily from 10,000 to 17,000 for the polycarbonate resin of the invention. The viscosity average molecular weight (Mv) is a value obtained in the same manner as in the first invention. For the adjustment of the molecular weight of the polycarbonate resin, phenol, p-tert-butylphenol, p-tert-octylphenol, p-cumylphenol and the like are used.

[0016] In this case, it is necessary that the amount of the terminating agent is adjusted to make the fraction of hydroxyl end groups of less than 7% by mole in the form of pellets described later.

The polycarbonate resin will be described under assumption where it is obtained by the solution process.

[0017] A polycarbonate solution after polycondensation is washed for purification in various kinds of known methods, and the purified polycarbonate solution is then pelletized in various kinds of known methods.

In the case where the free total phenol content of the polycarbonate resin obtained in the foregoing process is large, it is preferred that an elution treatment is carried out by using a solvent, such as acetone, dioxane and the like, to elute free total phenol, so as to make it to 80 ppm or less. In this case, in the form of pellets described later, the elution treatment is carried out in such a manner that the volume average molecular weight is from 10,000 to 17,000, the fraction of hydroxyl end groups is less than 7% by mole, and the free total phenol content is 80 ppm or less.

[0018] A releasing agent and, depending on necessity, from 20 to 100 ppm of a phosphorous antioxidant are added to the dried polycarbonate resin flakes after the elution treatment, and it is then pelletized by an extruder. It is necessary that the releasing agent in the pellets is from 100 to 500 ppm (preferably from 150 to 350 ppm).

When the releasing agent is less than 100 ppm, occurrence of defects due to flash caused by releasing failure is increased, whereas when it exceeds 500 ppm, defects of polarization and white turbidity as an optical disk are liable to be occur, and thus

both cases are not preferred.

[0019] As the releasing agent, a polyhydric alcohol fatty acid ester is preferably used, and examples thereof include a partial ester of a trihydric alcohol, such as glycerin, trimethylpropane, hexanetriol and the like, and a tetrahydric or more valence of alcohol, such as pentaerythritol, mesoerythritol, xylitol, sorbitol and the like, with a fatty acid having a carbon number of from 10 to 30. Examples of the fatty acid include capric acid, undecanoic acid, lauric acid, tridecanoic acid, myristic acid, pentadecanoic acid, palmitic acid, margaric acid, stearic acid, nonadecanoic acid, eicosanoic acid, behenic acid and the like. Specifically, a glycerin monoester, such as glycerin monostearate, glycerin monopalmitate, glycerin monomyristate, glycerin mono laurate and the like, pentaerythritol distearate, pentaerythritol tristearate, pentaerythritol monopalmitate, pentaerythritol dipalmitate, mesoerythritol trilaurate, xylitol trilaurate, xylitol distearate, xylitol tristearate, xylitol tetrastearate and the like are used. These esters may be used singly or in combination of two or more of them.

[0020] Examples of the phosphorous antioxidant include a trialkyl phosphite, such as trimethyl phosphite, triethyl phosphite, tributyl phosphite, trioctyl phosphite, trinonyl phosphite, tridecyl phosphite, trioctadecyl phosphite, distearyl pentaerythryl diposphite, tris(2-chloroethyl)phosphite, tris(2,3-dichloropropyl)phosphite and the like; a tricycloalkyl

phosphite, such as tricyclohexyl phosphite and the like; a triaryl phosphite, such as triphenyl phosphite, tricresyl phosphite, tris(ethylphenyl) phosphite, tris(butylphenyl) phosphite, tris(nonylphenyl) phosphite, tris(hydroxyphenyl) phosphite and the like; a monoalkyldiaryl phosphite, such as 2-ethylhexyldiphenyl phosphite; a trialkyl phosphate, such as trimethyl phosphate, triethyl phosphate, tributyl phosphate, trioctyl phosphate, tridecyl phosphate, trioctadecyl phosphate, distearylpennterythyl diphosphate, tris(2-chloroethyl) phosphate, tris(2,3-dichloropropyl) phosphate and the like; a tricycloalkyl phosphate, such as tricyclohexyl phosphate and the like; a triaryl phosphate, such as triphenyl phosphate, tricresyl phosphate, tris(nonylphenyl) phosphate, 2-ethylphenyl diphenyl phosphate and the like; and the like. These may be used solely or in combination of two or more kinds of them.

[0021] As described in the foregoing, it is necessary that the polycarbonate resin in a form of pellets has a viscosity average molecular weight of from 10,000 to 17,000 and a fraction of hydroxyl end groups of less than 7% by mole. When the fraction of hydroxyl end groups is too large, defects due to flash frequently occur. It is preferred that the free total phenol content is 80 ppm or less from the standpoint of prevention of occurrence of defects due to flash.

When the viscosity average molecular weight is less than 10,000, the mechanical strength of the molded article is lowered, whereas when it exceeds 17,000, distortion remains in the molded

article due to shortage of flowability upon molding, so as to lower the optical characteristics, and therefore both the cases are not preferred.

[0022] The measurement methods of the impurity amounts in bisphenol A and the fraction of hydroxyl end groups and the free total phenol content of the polycarbonate resin are as follows.

(1) Impurity Amounts in Bisphenol A

A sample is dissolved in acetonitrile, and quantitative analysis is carried out with liquid chromatography by using a known substance as reference.

(2) Fraction of Hydroxyl End Groups (in the case where p-t-butylphenol is used as a terminating agent)

A sample is dissolved in heavy chloroform and measured for ¹HNMR. The proton amount A in the ortho position and the proton amount B of the butyl group of the p-t-butylphenyl group as an end group are obtained with respect to the OH group, and it is calculated by the following equation.

Fraction of hydroxyl end groups (% by mole) =

$$100 \times (A/2) / ((A/2) + (B/9))$$

(3) Free Total Phenol Content

It is the total amount of the phenols, such as the dihydric phenol of the raw material monomer, the monohydric phenol of the terminating agent and the like, and is analyzed by the following method.

(1) 10 g of a sample is put in cylindrical filter paper.

(2) 120 cc of acetone and 2 or 3 pieces of glass zeolite

are put in a flat-bottom flask.

(3) Soxhlet extraction is carried out. The extraction time in the case of pellets is 4 hours from the point where the first acetone reflux is completed.

(4) The acetone solution is set in a rotary evaporator, and acetone is distilled off and concentrated to dry.

(5) After drying at 105°C for one hour in a dryer, it is stood to cool to room temperature.

(6) The content is dissolved by adding 10 cc of methylene chloride.

(7) 50 cc of a 0.1 N sodium hydroxide aqueous solution is added and stirred with a stirrer for 15 minutes.

(8) The content is stood to be separated, and about 25 cc of the sodium hydroxide solution as the aqueous layer (upper layer) is collected and filtered with 5A filter paper.

(9) The total phenol content of the filtrate is measured by a spectrophotometer.

[0023] The optical disk substrate of the invention can be obtained by molding the polycarbonate pellets in an arbitrary method, such as the injection molding method, a compacting molding method, the extrusion molding method and the like. Among these, the injection molding method using a stamper is preferred.

The invention will be then described more specifically with reference to examples and comparative examples.

[0024]

[Example] Next, although an example and the example of comparison

explain this invention still more concretely, this invention is not limited at all by these examples.

(Example 1)

1,000 kg of phenol and 100 kg of acetone were mixed, to which hydrochloric acid was added to carry out a condensation reaction at 65°C for 4 hours. The reaction solution was distilled to recover hydrochloric acid, water and a certain amount of phenol from the top of the tower. The matter at the bottom of the tower contained 32.0% by mass of bisphenol A, 64.4% by mass of phenol and 3.6% by mass of other compounds. A phenol solution of the bisphenol A was allowed to stand for 2 hours under the condition where it was cooled to 55°C. The solution was in a form of slurry containing a phenol adduct of bisphenol A. It was then subjected to suction filtration with a strainer, and the resulting crystals were washed with phenol to obtain a crystal sample (a). The crystal sample (a) was melted at 165°C, and phenol was distilled off under reduced pressure to obtain bisphenol A (b). The impurity concentrations of the bisphenol A (b) were 1,200 ppm for 2,4-bisphenol A, 200 ppm for a cyclic IPP dimer and 160 ppm for trisphenol.

[0025]

To 100 kg of the phenol adduct of bisphenol A obtained in the course of the process for obtaining the crystal sample (a), 100 kg of phenol was added and dissolved under heating to 95°C. When the phenol solution of bisphenol A was allowed to stand for 2 hours under the state where it was cooled to 55°C, the solution

was in the form of slurry containing crystals of the phenol adduct of bisphenol A. It was then subjected to suction filtration, and the resulting crystals was washed with phenol to obtain a crystal sample (c). The crystal sample (c) was melted at 165°C, and phenol was distilled off under reduced pressure, it was cooled under stirring to obtain bisphenol A (d) in a form of flakes. The impurity concentrations of the bisphenol A (d) were 180 ppm for 2,4-bisphenol A, 10 ppm for the cyclic IPP dimer and 20 ppm for trisphenol.

[0026]

Polymerization was carried out by using the bisphenol A (d) under the following conditions.

(1) Preparation of Polycarbonate Oligomer

To 400 L of a 5% by mass sodium hydroxide aqueous solution, 60 kg of bisphenol A was dissolved to prepare a sodium hydroxide solution of bisphenol A.

Then, the sodium hydroxide solution of bisphenol A maintained at room temperature was introduced at a flow rate of 138 L/hr and methylene chloride was introduced at a flow rate of 69 L/hr to a tubular reactor having an inner diameter of 10 mm and a tube length of 10 m through an orifice plate, to which phosgene was blown at a flow rate of 10 kg/hr, so as to carry out a reaction for 3 hours in a continuous manner. The tubular reactor used herein had a double tube structure, and cooling water is passed through the jacket part to maintain the discharging temperature of the reaction liquid to 25°C.

The pH of the discharged liquid was adjusted to from 10 to 11. The thus resulting reaction liquid was allowed to stand to separate and remove an aqueous phase, whereby a methylene chloride phase (220 L) was collected to obtain a polycarbonate oligomer solution.

[0027]

(2) Production of Polycarbonate

To 10 L of the polycarbonate oligomer solution obtained in the item (1), 118 g of p-t-butylphenol was dissolved, to which a sodium hydroxide aqueous solution (NaOH: 75 g, water: 1 L) and 1.17 mL of triethylamine were added, and stirred at 300 rpm at ordinary temperature for 30 minutes. 8 L of methylene chloride and a sodium hydroxide aqueous solution of bisphenol A (bisphenol A: 607 g, NaOH: 320 g, water: 5 L) were then added thereto and stirred at 500 rpm at ordinary temperature for one hour. Thereafter, 5 L of methylene chloride was added and stirred at 500 rpm at ordinary temperature for 10 minutes. After termination of stirring, standing separation was carried out to obtain an organic phase. The organic phase was alkali-washed with 5 L of a 0.03 N sodium hydroxide aqueous solution, washed with 5 L of 0.2 N hydrochloric acid, and washed with 5 L of water (twice) in this order, and then methylene chloride was distilled off to obtain polycarbonate in a flake form. The resulting polycarbonate flakes were dried in vacuum at 120°C for 48 hours, so as to obtain polycarbonate flakes having a viscosity average molecular weight of 14,500. About 50 kg of polycarbonate flake

was obtained in the similar operations.

[0028]

To the resulting polycarbonate in a flake form, 300 ppm of glycerin monostearate as a releasing agent and 40 ppm of tris(2,4-di-t-butylphenyl)phosphite as a phosphorous antioxidant were added, and then pelletized with an extruder. The pellets had a fraction of hydroxyl end groups of 4% by mole, a free total phenol amount of 40 ppm and a glycerin monostearate content of 280 ppm. The pellets were fed to an injection molding machine (DISK5, produced by Sumitomo Heavy Industries, Ltd.), and 600 pieces of disk substrates having a diameter of 130 mm and a thickness of 1.2 mm under the following conditions.

Cylinder temperature: 325°C

Metallic mold temperature: 90°C (stamper side)/85°C

Stamper: for CD-ROM

The resulting disk substrates were examined with a flaw defect inspection machine, and as a result, the defects due to flash were 4.5%. The disk substrates were subjected to accelerated deterioration under constant temperature and humidity of 90°C and 90% for 300 hours, and the block error rate was 5 when they were measured with an electric characteristics inspection machine.

[0029]

(Comparative Example 1)

Polymerization was carried out in the same manner as in Example 1 by using the bisphenol A (d) produced in Example 1 to

obtain polycarbonate flakes having a viscosity average molecular weight of 14,500. To the resulting polycarbonate in a flake form, 30 ppm of glycerin monostearate as a releasing agent and 40 ppm of tris(2,4-di-t-butylphenyl)phosphite as a phosphorous antioxidant were added, and then pelletized with an extruder. The pellets had a fraction of hydroxyl end groups of 4% by mole, a free total phenol amount of 40 ppm and a glycerin monostearate content of 20 ppm. 600 pieces of disk substrates were produced by using the pellets in the same manner as in Example 1. The resulting disk substrates were examined with a flaw defect inspection machine, and as a result, the defects due to flash were 11.0%.

[0030]

(Comparative Example 2)

Polymerization was carried out by the phosgene method using the bisphenol A (d) produced in Example 1 to obtain polycarbonate flakes having a viscosity average molecular weight of 14,500. To the resulting polycarbonate in a flake form, 600 ppm of glycerin monostearate as a releasing agent and 40 ppm of tris(2,4-di-t-butylphenyl)phosphite as a phosphorous antioxidant were added, and then pelletized with an extruder. The pellets had a fraction of hydroxyl end groups of 4% by mole, a free total phenol amount of 40 ppm and a glycerin monostearate content of 560 ppm. 600 pieces of disk substrates were produced by using the pellets in the same manner as in Example 1. The resulting disk substrates were examined with a flaw defect

inspection machine, and as a result, the defects due to flash were 4.0%, but after the disk substrates were subjected to accelerated deterioration under constant temperature and humidity of 90°C and 90% for 300 hours, the block error rate was 26 when they were measured with an electric characteristics inspection machine.

[0031]

(Comparative Example 3)

Polymerization was carried out in the same manner as in Example 1 by using the bisphenol A (b) produced in the course of Example 1 to obtain polycarbonate flakes having a viscosity average molecular weight of 14,500. To the resulting polycarbonate in a flake form, 300 ppm of glycerin monostearate as a releasing agent and 40 ppm of tris(2,4-di-t-butylphenyl)phosphite as a phosphorous antioxidant were added, and then pelletized with an extruder. The pellets had a fraction of hydroxyl end groups of 8% by mole, a free total phenol amount of 90 ppm and a glycerin monostearate content of 280 ppm. 600 pieces of disk substrates were produced by using the pellets in the same manner as in Example 1. The resulting disk substrates were examined with a flaw defect inspection machine, and as a result, the defects due to flash were 15.0%.

[0032]

[Effect of the Invention] According to this invention, the optical disk substrate which the defect in a flash becomes from

the polycarbonate resin and this resin which were suitable as a material of a few optical disk substrate can be offered, without increasing the addition of a release agent beyond the need.

[Document]

Abstract

[Abstract]

[Objects]

To provide a polycarbonate resin suitable as a base material of an optical disk substrate that faults due to burrs are low though the amount of a releasing agent to be added is not increased more than necessary and to provide an optical disk substrate consisting of the polycarbonate resin.

[Means for Solving the Problems]

This polycarbonate resin is manufactured by using 2,2-(4-hydroxyphenyl)propane containing 1,000 ppm or less of 2-(2-hydroxyphenyl)-2-(4-hydroxyphenyl)propane, 150 ppm or less of cyclic dimer of p-isopropenylphenol and 150 ppm or less of trisphenol compound as a raw material, has 10,000-17,000 viscosity average molecular weight and less than 7 mol% hydroxyl group terminal fraction and contains 100-500 ppm releasing agent.

[Selected Drawings] None